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ELECTRO-OPTICAL SYSTEMS, INC. Pasadena, California

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Report for 27 November 1961 - 27 February 1962

DEVELOPMENT OF LIGHTWEIGHT OPTICALLY ACCURATE REPLICA MIRRORS

Prepared for

U. S. Army Engineer Research and Development Laboratories Fort Belvoir, Virginia

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ABSTRACT

This progress report covers the quarter 27 November 1961 through 27 February 1962. The first program objective is to fabricate an astronomical quality 6" mirror by electroforming. Other mutually dependent objectives include electroforming bath evaluations, definition of optimum plating parameters, mirror surface coatings investigation, rigidizing structures studies, and extrapolation of results to the fabrication of large size, 18" minimum diameter mirrors.

The quarterly report summarizes the technical status, the technical progress details, and the second quarter plans. Since this report covers the entire quarter, there is some duplication of information presented in the letter report dated 7 January 1962.

This research is part of Project DEFENDER sponsored by the Advanced Research Projects Agency, Department of Defense.

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1. PROJECT TECHNICAL STATUS

During the first quarter, 27 November 1961 through 27 February 1962, progress was accomplished in many areas, including:

Stress instrumentation and measurement

Analysis procedures

Procurement and installation of electroplating equipment Anode-cathode geometric relationships

Rigidized thin film replication

Sensitizing layers

Rhodium post-plating coatings.

1.1 Masters

- a. Masters used for anode to cathode geometry studies were 3" diameter ground and polished Pyrex discs, 1" thick, and originally accurate to 1/4 wavelength. These were available from an earlier program.
- b. 6" spherical masters used for both thick and thin film replication studies were ground and polished BSC-2 convex masters, radius 14.188 ±.000120 inches and accurate to 1/4 wavelength. Two were fabricated during the quarter. One slightly damaged master was available from an earlier program.
- c. One 6" BSC-2 flat was ground and polished to an accuracy of 1/4 wavelength over the inner 4-1/2" diameter, with approximately 2 wavelengths of edge curl.
- d. Work is currently progressing on the fabrication of two more 6" BSC-2 spherical masters, 1/4 wavelength accuracy and 14.188 ±.000120 inch radius. Two 6" diameter BSC-2 flats are also in progress.

- e. Two 3" diameter Kanigen-plated steel flats have been secured and are ready for grinding and polishing.
- f. Machining has been initiated on both a 6" diameter spherical and a 6" diameter flat steel master which will be Kanigen plated, ground and polished.
- g. Five 3" x 3" x 1" square BSC-2 glass flats have been surface polished front and back and on two opposite sides to be used in determining stress phenomena in masters by photoelastic techniques during various phases of the replica process.

1.2 Master Attrition Studies

Two 3" square masters have been analyzed for residual stress in a polariscope.

1.3 Conductive Coatings

- a. The following conductive coatings have been tested:
 - i Immersion chemical silver Brashear process
 - ii Sprayed chemical silver
 - iii Vacuum deposited silver, one layer
 - iv Vacuum deposited silver, two layers
- b. Qualitative adhesion characteristics were obtained on the above coatings.
- c. Information was obtained on electroforming defects caused by pinholes.

1.4 Stress Phenomena

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- a. An extensive literature study was initiated to determine and understand the phenomena causing stress.
- b. Dr. Abner Brenner, National Bureau of Standards staff scientist and Spiral Contractometer inventor, was contacted.
- c. Dr. Joseph Kushner, Kushner Electroplating School and Evansville College, inventor of Stresometer was visited.

d. Temperature and current density effects on stress were measured and compared with reported data.

1.5 Stress Measurement Instruments

- a. The Spiral Contractometer, Stresometer, EOS strip test, and EOS strain gage strip were analyzed and compared (Table II).
- b. Stress tests were run with the EOS strip test and the Stresometer.
- c. Dr. Brenner indicated that the unmodified Spiral Contractometer does not meet our precision requirements.
- d. The EOS strain gage strip test has been shelved for the present.

1.6 Electroplating Baths and Bath Variables

- a. Four Barrett sulfamate nickel baths are operating.

 Each bath composition is standard except that the saccharin stress reducer content varies.
- b. A one-gallon bright-rhodium plating solution has been procured.
- c. Two acid-copper plating baths and one fluorosilicate chromium bath are also available.

1.7 Electroplating Equipment and Instrumentation

- a. The four nickel baths are contained in white Koroseal lined tanks, 28" long, 16" wide, with a working depth of 15". Two of these tanks have 8" wiers and sump areas used for dummying and locating the filters.
- b. Each tank has a Winscott D-10 cartridge filter for continuous filtration.
- c. One or moreLightning No. 30 mixers per tank are available for agitation.
- d. Tanks are controlled within $\pm 1/2^{\circ}F$. A portable temperature control accurate to $\pm 0.02^{\circ}F$ has just been procured.

- e. A Rustrak voltage recorder and an amperage recorder (each accurate to + 2 percent) are in use.
- f. A minimum of five power supplies are available:
 - i A 12-volt, 20-ampere rectifier with less than one percent ripple having a volt meter and 2 ampere meters (0 to 1 amps and 0 to 20 amps). The volt and ampere meters are accurate to ± 2 percent. An ampere-hour meter, which records the total ampere-hours plated and will shut off the current at a predetermined time, is included in the circuit.
 - ii Two 10-volt, 15-ampere rectifiers with less than 10 percent ripple
 - iii One 28-volt, 3-ampere rectifier; also used to
 produce 28-volt, 3-ampere superimposed AC
 current for superimposed AC studies
 - iv A 12-volt, 125-ampere rectifier with less than 35 percent ripple.
- g. Three ampere meters (one 0 to 500 ma, two (to 1000 ma) all accurate to $\frac{1}{2}$ 2 percent, are in use.
- h. The rhodium tank is a 2-1/2 gallon glass battery jar.
- i. Two separate ampere-minute meters are available.

1.8 Master Rotors and Fixtures

- a. Two 6" diameter master mounting assemblies were fabricated during the quarter.
- b. One lucite mounting assembly was constructed for the $30^{\prime\prime}$ masters.
- c. Three pivoted fractional horsepower rotor assemblies were constructed,
- d. A suitable anode holder for use in anode to cathode studies has been constructed.

1.9 Electroforming Environmental Control

- a. The analysis facilities and three of the four experimental tanks are located in a room air-conditioned to $70^{\circ}\mathrm{F}$.
- b. The laboratory floor is vacuumed once per working day.
- c. Laboratory access is limited to those involved in research electroforming.
- d. When not in use, all tanks are covered.

1.10 Chemical Analysis Procedures, Equipment and Chemicals

- a. Procedures have been established for nickel, boric acid, chloride, saccharin, pH, specific gravity, and surface tension analyses.
- b. Equipment and chemicals have been ordered for the saccharin analysis and procured for the other analyses.

1.11 Surface Coatings

- a. Surface coating studies have involved conductive coatings (1.3 above) and rhodium overcoatings on silver or nickel from which silver was stripped.
- b. Reflectivity and multi-layer surface coating studies on another program will be used to guide and augment surface coating studies on this program in the next quarter.

1.12 Materials Evaluation

During this quarter, the materials evaluation studies began with a literature survey.

1.13 Backing Structures for Rigidizing

a. Aluminum (with and without lightening holes) and BSC-2 glass backing structures have been used to rigidize the thin replicas electroformed from 3" optical flats.

- b. One 6" diameter BSC-2 concave backing structure has been fabricated.
- c. Four 6" concave aluminum backing structures have also been fabricated.

1.14 Optical Testing

- a. The thickness profiles and the replicas from the 3" optical flats have been tested by interference methods with another optical flat.
- b. The 6" replicas have been tested by a Ronchi test.

1.15 <u>Large Mirror Design and Extrapolation of Experimental</u> Results

 $\label{eq:continuous} \mbox{It was not logical to work on this phase in the first quarter.}$

2. TECHNICAL PROGRESS DETAILS

2.1 Background

Existing optical grinding and polishing techniques do not meet space age physical, environmental, or manufacturing requirements. See Table I for a comparison of reflective optics fabrication techniques. A major state-of-the-art breakthrough in meeting these requirements can potentially be achieved by electroformed replicas. Earlier work at Fort Belvoir, ERDL (Ref. 1) demonstrated the ability of electroforming to meet surtace accuracies and reflectivities required for searchlight mirrors. EOS has advanced the art of electroforming optical replicas by producing lightweight reflectors of subastronomical accuracy. The reflectivity of electroformed replicas has equalled or exceeded that of ground and polished glass, having equivalent coatings. This EOS work indicated that astronomical quality replicas could be produced by closely controlling electroforming parameters.

2.2 Objectives

The primary program objective is to fabricate a 6" astronomical quality mirror. Astronomical quality is defined as an angular error less than $\frac{1}{2}$ seconds. Mutually dependent program objectives include studying zero-stress mirror skins, surface coatings, optimum plating parameters, various electroforming baths, structural members for rigidity, and controls for replicating mirrors 18" diameter or greater.

2.3 Technical Approach and Attack Rationale

From previous work, five problem areas require intensive investigation:

2.3.1 Glass Master Attrition and Alternate Master Materials

Most electroformed optics have initially been made from glass masters. Glass master attrition has been high. For production runs, other materials might be advantageous for replica masters. But glass is still the most economical and accurate material for initial small size masters. Therefore, glass master attrition during electroforming should be studied. Since metal has many inherent physical and electrical advantages over glass, Kanigen plated metal masters will be compared with glass masters.

2.3.2 Residual Stress

This is the major problem in electroforming replica optics. For an aspheric optic even a very small amount of residual stress can be detrimental to accuracy. Current density variation over the replica during plating causes the major stress problem. Stress varies directly with current density, (Fig. 2-b). Stress variation problems during replication are shown in Fig. 1. Current density varies with solution throwing power, the nature of the conductive layer on the master and the local electric field strength as affected by edge conditions and surface curvature. If the current density over the entire replica can be maintained at a constant value and if the electroplating bath can be regulated such that there is zero stress at this value, a zero stress replica skin can be formed. To retain high replica accuracy the conductive layer must be very thin. This introduces resistance problems. To overcome resistance problems and other factors causing thickness variations, an anode to cathode geometry study is required. It is logical to compare the stress problems using Kanigen plated metal vs. glass masters.

Variations in agitation, temperature, stress reducer, wetting agent, bath efficiency, bath conductivity, bath composition, contaminants, pH, current supply, and crystal size in the deposit also affect stress. Careful analysis and instrumentation are required to study these variables.

Earlier work (Refs. 2 and 3) has shown that deposits less than .0001 in. thick are highly stressed, depending on the base metal material and crystal structure. As thickness increases, residual stress changes rapidly from an initial high tensile or compressive stress to an equilibrium stress which is a function of the bath variables. After electroforming stops, the residual stress can change slightly if the replica remains in the bath. The initial stress and the equilibrium stress values are the most significant. Though the initial stresses act on a very thin film, they are optically very important. Various conductive coatings will be studied to minimize the initial stress effects.

2.3.3 Accurate Stress Measurement

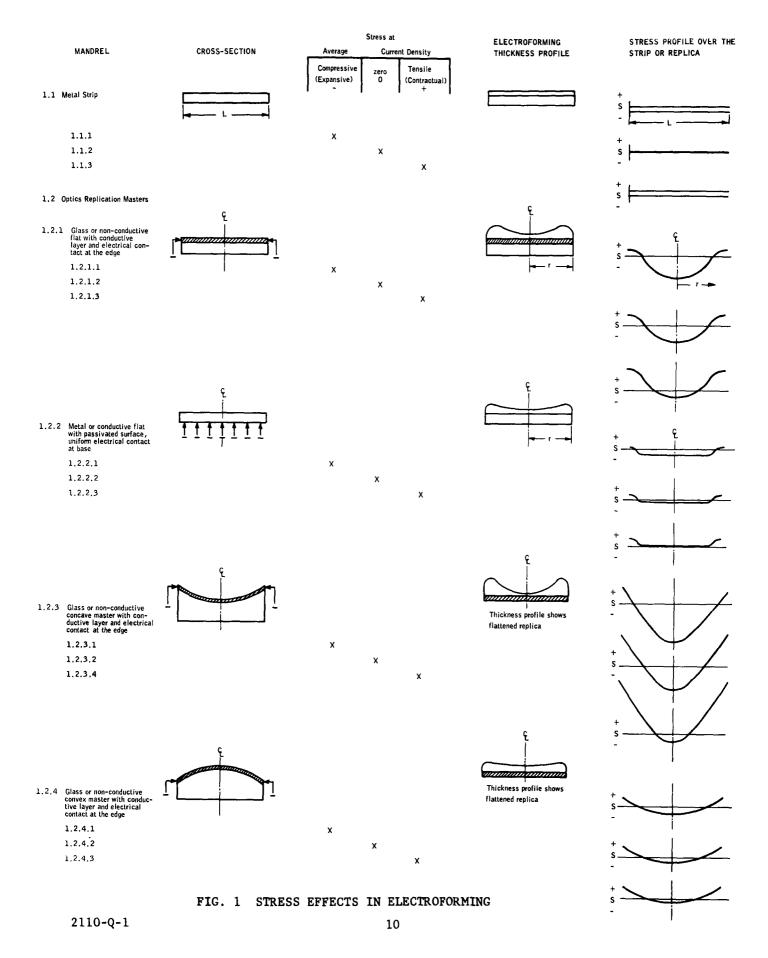
This is necessary to produce a zero stress skin. The optimum stress measurement instrument should be determined by tests and analysis. Table II summarizes four instruments. After choosing the optimum instrument, stresses measured with the instrument can be correlated with those in the replicas by noting the effects of stress on replica optical accuracy.

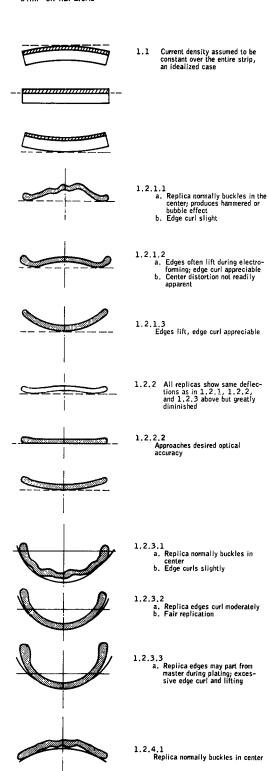
2.3.4 Surface Coatings

Vacuum deposited surface coatings over electroformed replicas have been inconsistent in previous work. In World War II searchlight fabrication electrodeposited rhodium was used. The electroforming of astronomical quality replicas depends in part on the ability to vacuum coat over a replicated surface or on a master before electroforming.

2.3.5 Structural Members to Provide Rigidity

Lightweight astronomical quality reflectors can only be faoricated using a supporting structure. Two replicating approaches are feasible: an all-electroformed, self-supporting replica and a thin electroformed replica attached to a backing structure. This backing structure could be electroformed. On this





1. a. Anodes are assumed to be of infinite distance and infinite area

 For comparative purposes the average current density and replica thickness is assumed constant for each replica

1.2.1 a. The varying thickness profile is caused by (a) the resistance of the conductive layer which varies almost inversely with the radius and (b) the electric field concentration at the edges in which the current density varies almost inversely with the square of $(\mathbf{G}_{\mathbf{c}}-\mathbf{r})$ where $\mathbf{G}_{\mathbf{c}}$ is the radius of the flat and \mathbf{r} is any radius

- to the edges in which the current certainty varies amounts inversely with the square of to-r where \(\mathbb{c}_i \) is the radius of the flat and \(r \) is any radius \(b \). Due to resistance and edge effects the thickness at any part will vary directly with the average current density and the radial distance from the centerline, i.e., as the average current density rises for the same average thickness the replica edge thickness will increase; at the center the thickness will decrease
- c. Edge effects can be reduced or eliminated by shields
- d. Conductive layer resistance effects can be eliminated by varying anode to cathode geometry

1.2.2 a. Conductive master with uniform electrical contact with base would eliminate conductive layer effects

b. Edge effects can be reduced or eliminated by shields

- 1.2.3 a. Current density affected by edge effects and conductivity plus master concavity, which increases the edge effect
 - Conductive master with no resistance, would reduce thickness variation effects as in 1.2.2 above
 - Theoretically a metal master with edge shields and a conforming anode would produce a uniform thickness and uniform current density, all other variables remaining constant

- 1.2.4 a. Master convexity reduces edge and conductive effects on current density and thickness
 - b. A conductive master as in 1.2.3b above would reduce conductive thickness variations
 - c. Theoretically a metal master with edge shields and a conforming anode would produce a uniform thickness and uniform current density, all other variables remaining constant

FIG. 1 STRESS EFFECTS IN ELECTROFORMING (CONTINUED)

1.2.4.2 Little or no edge curl or lifting. Fair replication

1.2.4.3 Edges lift and curl moderately program, both approaches are being investigated concurrently. Thin rigidized replicas can demonstrate the ability to replicate astronomical quality optics and can at the same time generate data necessary to fabricate thicker replicas. If attachment problems are solved, the thin film approach is more advantageous than thick allelectro formed replicas.

2.4 Technical Results

2.4.1 Masters

Methods for fabricating glass masters are well known. Glass masters can be made with high accuracy. Therefore, master construction was initiated first using BSC-2 glass, which has high optical quality and a thermal expansion coefficient comparable to nickel. A concave spherical test plate (radius 14.188") and a matching convex master from a previous program were available. Two additional convex spherical masters were produced using this test plate. These are accurate to 1/4 wavelength. Front and back edges have been beveled and polished to prevent edge attrition. The back surfaces have been ground and polished to allow observation of the replica surface prior to parting. Two more 6" convex masters are in process. The 6" diameter flats are also beveled and polished similarly. One 6" flat has been completed, two are in process.

Several 3" diameter optical flats accurate to 1/4 wavelength are also available. They are more economical to use because several can be vacuum coated simultaneously. They provide much data on anode to cathode geometry, replication techniques, and surface coatings. The backs are also ground and polished on these masters.

Two Kanigen plated 3" flats are now ready for optical polishing.

2.4.2 Master Attrition Studies

Master attrition on this program has not been a serious problem. However, previous replication work at EOS and elsewhere has indicated serious master breakage problems under conditions which have not heretofore been predictable. Because of the expense of glass masters, positive methods are essential for preventing breakage and minimizing gradual surface degradation.

Glass master attrition indicates that the following conditions can cause problems:

- a. Thin Masters As the master diameter to thickness ratio increases, rigidity decreases for a constant diameter. A ratio of about 6 or less is considered acceptable for astronomical accuracy. With masters having higher ratios proportionately more gross breakage can be expected.
- b. Old Masters Old masters with residual stress may have limited strength. Previous stresses can reduce the fatigue life and tensile strength. Strength reduction varies with the amplitude and duration of previous stress (Ref. 4).
- c. <u>Poor Surface Qualities</u> Rough glass has a much lower yield point than highly polished glass. Minute cracks and roughness points act as crack growth centers.
- d. Sprayed Silver Conductive Coating Applied on Sensitized Glass - A spray silver conductive coating involves three factors which increase glass attrition:

Strong acids and stannous chloride used in cleaning and activating produce roughness.

- ii If absorbed by the glass, water used during coating can later promote cracking (Ref. 5).
- iii The adhesion of chemically deposited silver to glass can cause cracking and chipping problems during parting.
- e. Water on the Glass and High Localized Stress

 During Parting Water or water vapor at the glass interface during parting, drastically lowers glass tensile strength. When stress is present water combines with silicon dioxide at a crack center to form silicon hydroxide.

 Silicon hydroxide formation on each side of the crack increases the crack growth. Therefore, both water and high parting stresses must be minimized.

To reduce attrition we have concentrated on vacuum deposited conductive coatings. Pinholes in these coatings cause electroforming problems. But vacuum coatings have low adhesion to glass and can be deposited with very little variation in thickness.

Two 3" square BSC-2 masters analyzed in a polariscope show polishing and grinding stresses concentrated largely at the corners. This analysis has been limited, because available polariscopes show stress interference patterns only at extinction. With suitable polaroid films the intervening stress colors can be delineated.

2.4.3 Conductive Coatings

Several types of conductive coatings have been investigated including:

Sprayed chemically deposited silver
Immersion silver by the Brashear process
Vacuum deposited silver - one layer
Vacuum deposited silver - two layers

Because of improved replica quality and reduced master attrition, vacuum deposited conductive coatings are now being used exclusively on this program. A single-layer coating usually has an excessive number of pinholes and results in premature parting and replica wrinkling, because bath solution seeps between the conductive layer and the master surface. Two layers of vacuum deposited silver have produced good results (Table V). Between layers, the master is rotated 90° to allow a different angle of impingement of the silver.

Comparing initial stress produced on a brass base with that produced on a brass base with a vacuum deposited silver coating (Table IV) indicates that the base material has an appreciable effect on the initial stress. This confirms previous reports (Refs. 2 and 3).

Vacuum deposited conductive coatings are superior to chemically deposited coatings in regard to ultimate replica surface quality. The difference is noted when the silver coatings are removed from the replica surface. Since the vacuum deposited coatings are thinner and of more uniform thickness, the silver surface on which the replica builds up (the surface away from the master) is smoother and more reflective than for chemically deposited coatings. Therefore the surface of the nickel replica, which is a faithful copy of the silver surface, is also smoother and more reflective. Furthermore, since vacuum deposited coatings are more easily dissolved than chemically deposited coatings, the stripping solution has less chance to attack the nickel.

2.4.4 Stress Phenomena

Many factors cause stress in electrodeposits; various theories have been proposed. To accomplish the program goals, it is necessary to understand the variables affecting stress. An extensive literature study of the subject has been initiated and visits have been made to two leading electrochemical stress experts: A. Brenner, National Bureau of Standards; and J. Kushner, Kushner Electroplating School and Evansville College. Stress theories will be summarized in the final report and related to optics replication.

EOS data correlates the linear log stress versus temperature curve proposed by J. Kushner (Fig. 2-a). For current densities above 5 amps/ft 2 the curves shown in Fig. 2-b are typical.

2.4.5 Stress Measurement

Electroplating stress has traditionally been measured by a metal strip plated on one side and allowed to deflect during or after plating. The EOS strip and the EOS strain gage stress instruments are simple strip tests. (See Fig. 1 for an explanation of the deflection of plated strips.) In the Brenner-Senderoff Spiral Contractometer a strip is wound in a spiral and connected to a pointer (Ref. 6). J. Kushner's Stresometer correlates stress with volume variations produced by a deflecting disc (Ref. 7). These instruments are compared in Table III.

Initial stress tests were performed with the EOS strip test. Efforts to produce a uniform plating thickness by shielding were not encouraging. Therefore, use of the strip test was discontinued when the Stresometer arrived. The EOS strain gage strip test inherently has thickness problems similar to the simple strip test and had not been sufficiently developed to be regarded as dependable. Dr. Brenner indicated that the Spiral Contractometer did not have the precision desired for this program. Extensive modifications to the Spiral Contractometer have produced precise measurements but these modifications are expensive and do not eliminate other problems (Ref. 8).

Initial tests with the Stresometer have been run with thin shim-stock discs of 2-, 3-, and 5-mil thickness. Erratic calibration and test behavior of 2- and 3-mil discs, shown in Table III, could not be completely explained. Therefore, the 5-mil shim disc was standardized for testing at this time. The reproducibility of the Stresometer is better than \pm 3 percent, as indicated in duplicate tests 9 and 11, 12 and 14, and 15 and 16.

Because of its design, the Stresometer is very temperature sensitive and acts as a thermometer. Residual stress is also highly affected by temperature variations. Therefore, a controller accurate to $\frac{1}{2}$.02°F was purchased and installed.

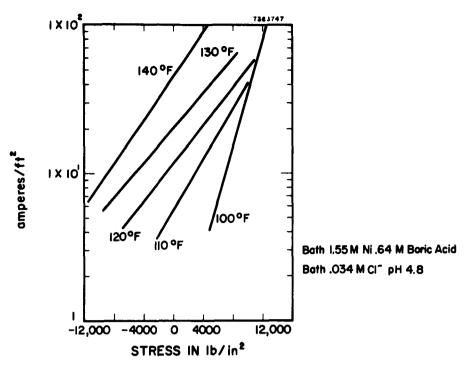
2.4.6 Electroplating Baths and Bath Variables

Work in this quarter has concentrated almost entirely on sulfamate nickel electroforming. Sulfamate nickel has excellent strength and corrosion-resistance characteristics. It is suitable for production of replica masters, and it can be plated in tensile, zero, or compressive stress. Related EOS programs have contributed much pertinent data to this program on the subject of sulfamate nickel plating stress.

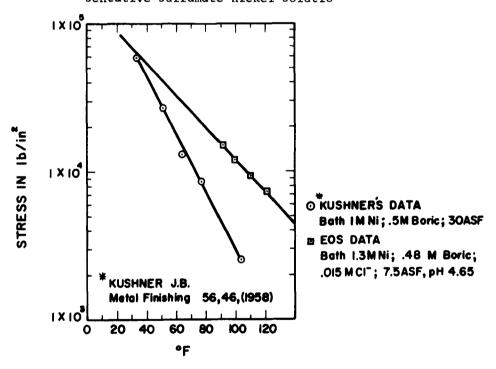
Standard Barrett sulfamate nickel baths with varying saccharin stress reducer concentration are used. Other proprietary baths and stress reducers could be tried, but all are basically similar and no major difference in results would be expected. It is probably more valuable to thoroughly study one bath than to briefly try several.

The rhodium solution was tried only as a means of providing corrosion-resistant reflective post-electroforming coating.

By plating thin replicas on optical flats having predetermined surface profiles, the anode to cathode geometry and shield effects can easily be studied. The replica thickness profile is measured by optical interference methods. Using this profile, along with the average replica thickness and surface geometry of the master; the percentage thickness variation over the entire replication

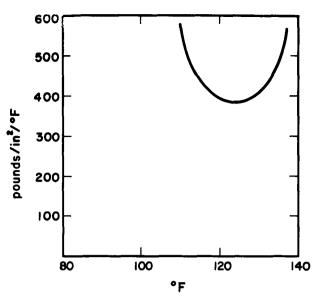


2a Current density vs stress at constant current density for a representative sulfamate nickel solution



2b Stress vs temperature at constant current density for representative sulfamate nickel solutions

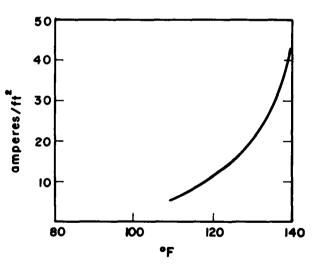
FIG. 2 STRESS VARIATION WITH CURRENT DENSITY AND TEMPERATURE



500 500 500 80 100 120 140

- 2c Stress change with temperature vs temperature at constant current density and zero stress **
- 2d Stress change with current density vs temperature at zero stress **

2e Current density vs temperature at zero stress **



2f Stress variation within ±2.5 percent of zero stress current density vs temperature **

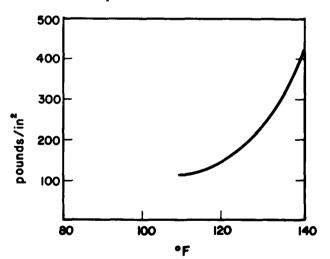


FIG. 2 STRESS VARIATION WITH CURRENT DENSITY AND TEMPERATURE (CONTINUED)

** Data derived from Fig. 2a

2110-Q-1

can be plotted against the radius. The anode-cathode geometry relationships, data and thickness profiles are shown in Fig. 3. Tests 2-7, -8, -9, -13 and -14 of Fig. 3, show that thickness variation decreases as average thickness increases. They also demonstrate the effects of current density decrease at the edges caused by gas collection and shielding. In these tests the mounting fixture face plate collected gas which partially shielded the edge from the anodes. Tests 2-19, -20, -29 and -30 show the edge effects when gases were vented, and anode shielding was changed by introducing another anode.

Tests 2-19 and -21 show a $\frac{1}{2}$ 6 percent thickness variation, which approaches the desired variation necessary to produce near-zero stress skins. Figs. 2-c, -d, -e, and -f show stress variation with temperature and current density; the zero stress current density at constant temperature; and the stress variation caused by a 5 percent spread in current density at the zero stress point at various temperatures for a typical sulfamate nickel bath. Figs. 2-c, -d, -e, and -f are derived from Fig. 2-a. Stress measurement instrument analyses and these figures indicate that temperature variations less than 0.1°F and current density variation over the plating replicaless than $\frac{1}{2}$ 2.5 percent are necessary to produce replica stress lower than the precision obtainable from the best stress measurement instruments.

Stress reducer variations are being studied. From Fig. 2 the most desirable stress reducer concentration would produce a zero stress current density between 5 and 10 amps/ft 2 at as high a bath temperature as practical but below $140^{\circ} F$. Unfortunately, high bath temperatures produce thermal expansion problems between the replica and master. For this reason, plating temperature has been kept between 90 and $100^{\circ} F$.

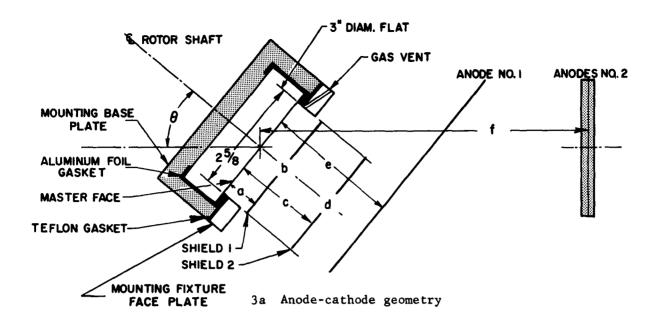
Previous work by V. J. Marchese indicated that AC superimposed over DC in nickel chloride plating baths reduces stress (Ref. 9). Superimposed AC versus DC tests are summarized in Table IV.

Because the test plating thickness was thin, results cannot be called conclusive. In each comparative test, the superimposed AC runs gave stresses considerably higher than without AC. Since these tests reproduced well, superimposed AC tests were discontinued. This failure to reduce stresses can probably be explained by comparing the chloride and sulfamate ions. Chloride ions have a high attraction, coordination value for other ions. This coordination power probably explains the fact that stress increases with increasing chloride ion concentration. Therefore, AC must effect the chloride ion coordination power. Also, chloride ions assist in dissolving nickel anodes. Anodes corrode poorly in pure sulfamate baths. It can be concluded that superimposed AC, which makes the master anodic intermittently, might have little effect on the stress mechanism in sulfamate baths. Our tests confirm this.

Brenner reports that agitation reduces stress. Ultrasonics are the ultimate in agitation. A 90-KC, 500-watt ultrasonic agitation unit with 6 transducers was evaluated by means of the EOS strip test. In very case the stress produced during ultrasonic agitation was greater than with normal mixer agitation. Perhaps the ultrasonic energy increased the strain energy in the deposit at this particular frequency. Since the stress was greater than with normal agitation, ultrasonic tests were discontinued. Time did not permit evaluations at other frequencies.

2.4.7 Electroplating Equipment and Instrumentation

Continuous filtration, precise control and standardized conditions have been the guide for equipment and instrumentation design and procurement. Foreign particles can produce localized stress concentrations when deposited on electroforming replicas. These are removed by continuous filtration. Two tanks have constant level control by using a weir and sump. The sump serves as a dummying cavity and as a location for the tank filter.

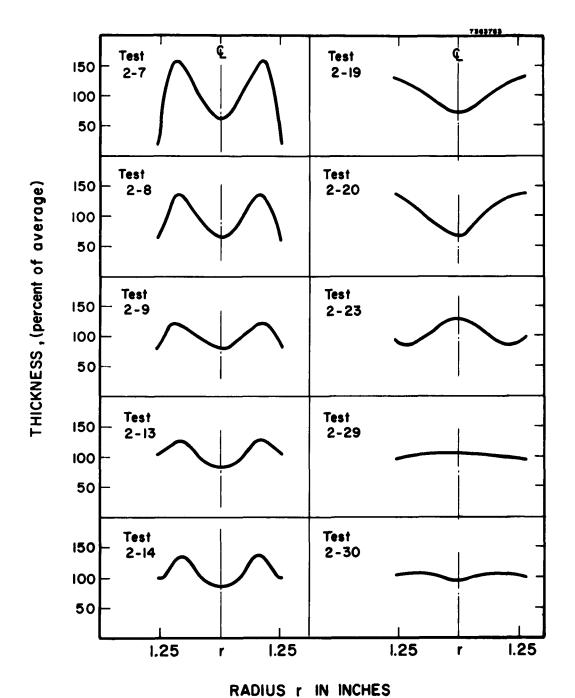


3b Anode-cathode geometry dimensions

											7363762
Test No.	Thickness in .001 "	θ	a (inches)	b (inches)	C (inches)	d (inches	e (inches	Anode No. I	Gas Vent	f	Anodes No. 2
2-7	.025	45°							NO	14 ½	3-9"LG 3 2 ovals
2-8	.071								NO		u
2-9	.103	11							NO	"	ıı
2-13	.366	11							NO	**	11
2-14	.674	11							NO	"	11
2-19	.382	11					2 1/2	25 Nickel disc	YES	_	_
2-20	.329	11					21/2	25 Nickel disc	YES	_	-
2-23	.678	11			-	34	_		YES	141/2	2-9"LG 3½ ovais
2-29	.857	11	5 8	2 	15	34	21/2	2×3 Strip	YES	"	11
2-31	1.12	-	5	2 🛔	2	34	21/2	2x 3 Strip	YES	11	10

FIG. 3 SUMMARY OF REPLICA THICKNESS PROFILES VERSUS ANODE-CATHODE GEOMETRY

2110-Q-1



3c Thickness profiles in percent of average versus radial distance for 2-5/8" replicas

FIG. 3 SUMMARY OF REPLICA THICKNESS PROFILES VERSUS ANODE-CATHODE GEOMETRY (CONTINUED)

A voltage recorder and amperage recorder plus three available ampere-minute meters enable tests and tanks to be monitored continously for better control. A superimposed AC current plus DC rectifier assembly and two amp meters have been assembled.

2.4.8 Master Rotors and Fixtures

Electrochemical reactions vary with solution depth. This variation is caused by hydrostatic pressure on the electrode films, electric field effects between the cathode and anode, solution stratification, gassing effects, etc. Therefore, symmetric optics must be gradually rotated for uniform plating. Three fractional horsepower master rotor assemblies and supporting master holding fixtures have been assembled to provide continous duty. Adequately engineered equipment for electroforming replica optics cannot be over-emphasized.

2.4.9 Electroforming Environmental Control

An air conditioned room and adequate dust control have been provided to minimize environmental problems causing stress.

2.4.10 Chemical Analysis Procedures, Equipment and Chemicals

Procedures and EOS equipment have been set up to provide the necessary chemical analysis. Analyses are now being tabulated to provide control guides.

2.4.11 Surface Coatings

During the first quarter, coating studies were primarily concerned with the conductive coatings. In the remainder of the program more work will be done in developing reflective coatings for electroformed replicas. Some allied exploratory work in replica coatings is also being conducted on another contract. The results will be available for use on this program.

Procedures for stripping conductive silver layers and subsequent rhodium plating have been developed. A

summary of rhodium-plated replicas is given in Table VII. To achieve maximum brightness, replicas must be plated at current densities above 30 amps/ft² with little agitation. Efforts to rhodium plate over silver were not promising and have been discontinued. Also silver can contaminate rhodium baths and has presented corrosion and adhesion problems in reflectors.

2.4.12 Material Evaluation

Final report will summarize this phase.

2.4.13 Backing Structure for Rigidizing

During this quarter, replica optics were rigidized with both aluminum and BSC-2 glass backing structures. Glass backing structures were initially used in order to minimize thermal expansion problems. Replicas having metal backing structures initially suffered from poor adhesion between the nickel replica and the bonding material. This was caused by improper cleaning procedures. Suitable methods for cleaning the nickel prior to the adhesive application were developed. Aluminum backing structures, with and without lightening holes, have also been used with good results. Replication accuracies using both glass and aluminum backing structures have been as good as 1/4 wavelength over 2 inches. Replicas made during this quarter included 2.0" diameter flats and 2.5" diameter flats.

The approach described above (electroformed replica skin bonded to a metal backing structure) involves the use of organic adhesives. However, these adhesives are completely encapsulated in metal and should be well protected from the space vacuum and radiation effects which may be detrimental to plastics. It should be noted that organic adhesives have been used successfully in the space environment in solar cell panel components.

During the remainder of the program the basic electroforming data now being accumulated should make possible the fabrication of high-quality all-electroformed replicas.

2.4.14 Optical Testing

Work in this quarter used optical flats only. No 5-5/8" astronomical quality spherical replicas were made. (However, two 5-5/8" spherical replicas were made prior to the submission of this report. They were evaluated by means of the Ronchi test.)

2.4.15 <u>Large Mirror Design and Extrapolation of Experimental</u> Results

Final report will summarize this phase.

3. PLANS FOR NEXT QUARTER

3.1 Masters

The two BSC-2 spherical masters, the one BSC-2 6" flat, the Kanigen plated steel flat and the Kanigen plated steel spherical master will be completed. No additional masters are contemplated at this time.

3.2 Master Attrition Studies

The polariscope as a tool for analysis of residual stress and stress during various electroforming operations will be studied in greater detail. Since the measurement of strains in the glass masters is an accurate stress indicator optical flats having both front and back surfaces accurate within 1/4 to 1 wavelength will be analyzed for strains.

3.3 Conductive Coatings

- a. Studies will continue using vacuum deposited silver in two layers.
- Major effort in this area will be toward developing
 a suitable alternate conductive layer to silver.
 Copper, nickel and gold will be attempted.
- c. If feasible, quantitative information will be obtained regarding adhesive characteristics by the use of a high speed centrifuge.
- d. The relationship of conductive coating thickness to stress and electrical conductivity will be investigated.

3.4 Stress Phenomena

A literature study and various stress theories will be analyzed and summarized.

3.5 Stress Measurement Instruments

Unless future tests dictate another choice, the Stresometer will be used almost entirely for stress regulation and control.

Continuous manual control of stress by correlating and regulating the Stresometer tests will be tried.

3.6 Electroplating Baths and Bath Variables

Work will continue on the study of all variables effecting stress. Current density, anode to cathode geometry, temperature, agitation, and stress reducer concentration, will receive the major attention.

Sample replications using copper plating baths and possibly chrome and iron will be attempted.

Electroplating baths and bath variables will be summarized as a guide to optics replication.

3.7 Electroplating Equipment and Instrumentation

If necessary instrumentation will be constructed to regulate current more precisely than is now possible. This might require only constant rectifier input voltage control.

If necessary, finer filter cartridges will be procured to eliminate sub-micron particles.

3.8 Master Rotors and Fixtures

As dictated by anode to cathode geometry studies, additional anode fixtures will be constructed.

3.9 Electroforming Environmental Control

Shoe dust will be eliminated by a foot bath. If sub-micron particles are a problem, a dust hood will be constructed for one tank. No humidity or temperature problems are contemplated.

3.10 Chemical Analysis Procedures, Equipment and Chemicals

All analysis procedures will be in operation. If the saccharin analysis cannot be accomplished by a volumetric technique, spectrographic methods may be studied.

3.11 Surface Coatings

Surface coatings will include the following, silicon monoxide, aluminum, chrome, copper, nickel, gold, and magnesium fluoride. Surface coating adherence and environmental resistance will receive major emphasis. The ability to electroform over multiple coatings will also be studied.

3.12 Materials Evaluations

 $\label{eq:Based on program tests and surveys, an electroforming} \\ \text{materials summary will be written.}$

3.13 Backing Structures

For thin replicas, backing structures will be made progressively lighter to obtain optimum rigidity and lightness consistent with astronomical accuracy. Milled and honeycomb structures will be tried.

For thicker, all-electroformed mirrors, torus, ring, or monocoque backing structures will be grown in place.

3.14 Optical Testing

A Ronchi test specifically designed for fast spherical mirrors should be available soon for better optical testing. Checking thickness profiles and flat replicas will continue using optical flats.

3.15 Large Mirror Design and Extrapolation

Program evaluations will be extrapolated to provide design criteria for astronomical reflectors, 18" or larger.

4. SUMMARY AND CONCLUSIONS

Work in this quarter demonstrated that thin rigidized, electroformed, replicas of at least 3" diameter can be made with surface
accuracies sufficient to satisfy the objectives of this contract.

Problems in electroforming over vacuum deposited conductive coatings
have been solved. Criteria for bath temperature and current density
control were determined and implemented. The stress phenomena and
stress measurement instruments were analyzed. Stress measurement has
been standardized and precise stress measurements are being run.

Replica thickness variation studies are providing the basic design information required for more accurate replication to be accomplished in the second quarter. Rhodium coating tests have resulted in standardized procedures for rhodium plating replicas.

Masters, rotors, fixtures, and plating equipment are all available or under construction.

No program delays are contemplated.

REFERENCES

- The Engineer Research and Development Laboratories, The Engineer Center, Fort Belvoir, Virginia, "Manufacturing Process for Standard 60" Reflectors," Proceedings American Electroplaters Society, 34, 122, 1947.
- 2. V. Zentner, A. Brenner, and C. W. Jennings, "Physical Properties of Electrodeposited Metals, I Nickel," American Electroplaters Society Research Report, Serial No. 20.
- 3. J. B. Kushner, "The Role of the Basis Metal in the Production of Stressed Electrodeposits," Proceedings American Electroplaters Society, 45, 28 (1958).
- 4. E. B. Shand, "Experimental Study of Glass: I, The Fracture Process,"

 Journal of the American Ceramic Society, 37, 52, (1954).
- 5. R. E. Mould, "Strength and Static Fatigue of Abraded Glass Under Controlled Ambient Conditions: IV, Effect of Surrounding Medium,"

 Journal of the American Ceramic Society, 44, 481, (1961).
- 6. A. Brenner and S. Senderoff, "A Spiral Contractometer for Measuring Stress in Electrodeposits," Proceedings American Electroplaters Society, 35, 53, (1948).
- 7. J. B. Kushner, "A New Instrument for Measuring Stress in Electrodeposits," Proceedings American Electroplaters Society, <u>41</u>, 188, (1954).
- 8. H. Watkins and A. Kolk, "Measurement of Stress in Very Thin Electrodeposits," Journal of the Electrochemical Society, 108, 1018, (1961).
- 9. V. J Marchese, "Stress Reduction of Electrodeposited Nickel,"
 Journal of the Electrochemical Society, 99, 39, (1952).

TABLE I COMPARISON OF REFLECTIVE OPTICS FABRICATION TECHNIQUES

		Optical Pr	operties		Physic	al Properties	
Technique	Material	Present ability to meet 1 \(\lambda\) accu- racy or better	Surface Quality	Stability with time and temp	Weight	Ability to with- stand mechanical impact, vibration	Ther tivi Comp
Grinding					[l
1	Glass	Yes	1	1	8	8	
2	Glass with lightweight backing	Yes	2	5	5	7	
3	Metal	Yes	8	3	7	2	
4	Kanigen plated metal	Yes	6	4	6	4	
Replication					···		
5	Plastic	No	7	8	4	6	
6	Plastic with con- forming backing	Yes R and D	5	7	3	5	
7	Thin electroformed replica with back-ing sandwiched on	Yes R and D	3	6	2	3	
8	Electroformed replica	R and D	4	2	1	1	

Notes: 1. Rankings are estimates based on existing technology and are ranked from 1 the most acceptable to 8 the least

2. Rankings will be modified in the final report by including data derived both from this contract and existing

Adaptability to Space Environment

Manufacturing and Storage Properties Criteria

		Dill I I V C I I I C I C							
1 on	Thermal Conduc- tivity of Completed Mirror	Radiation Resistance	Vacuum	Producti 1 mirror	on Time 10 mirrors	Master time use/replication	Storage and Problems	Cos 1 mirror	t 10 mirrors
	7	1	1	1	5		8	1	5
	8	2	5	2	6		7	3	6
	•			_	•		,	5	٥
	3	3	3	7	8 7		6	8	o 7
	4	,	4	,					
	6	8	8	3	3	3	5	2	2
	5	7	7	4	1	1	4	4	1
	2	6	6	5	2	2	3	6	3
	1	4	2	8	4	4	2	7	4

B the least acceptable.

2

nd existing technology.

TABLE II

COMPARISON OF STRESS MEASUREMENT INSTRUMENTS

					Instrument S	tress Sensit	ivity at Con	3 ta
Instruments and Description	Base Mat'l. (3)	Base Thickness	Surface Finish	Base Expendability	Plating Thick- ness for Com- parison 10 din.	Unit of M	easurement	Ba Th
1.Spiral Contractometer (Brenner-Senderoff) Vertical Spiral (1)	SS Cu	.010030 .010030	Any finish possible	Reusable expendable	5 ± .15	<u>+</u> 1°	Dial	. (
2.Stresometer (2) (Kushner) 1635: capillary ratio horizontal disc	SS Cu Br Films	.00	Any finish possible	Reusable Expendable Expendable Expendable	5 ± .15	± .005" ± .005"	Capillary Height	.0
3.Strip test (EOS) Vertical Strip	ss	.010	200 grit Emery	Reusable	5 +4 5 - 1	± .005"	End Deflection	0.
4.Strip test with strain gage (EOS) Vertical Strip	Ni passi- vated	.008	2 RMS	Reusable	5 +4	+ .000001"	Strain of fibers on back of plated strip converted from voltage	.0

Notes:

- (1) Tabulated from Bibliography Ref. 6
- (2) Bibliography Ref. 7
- (3) SS = Stainless steel; Cu = Copper; Br = Brass shim stock; Films = vacuum coatings over base material; Ni passivated = passivated electroformed nickel
- (4) A. Brenner Private Conversation held January 16, 1962.

TABLE II - (CONT'D.)

ivity at Con	stant Ten	perature	Temperature				1	
easurement	Base Thick.	Stress lb/in ²	Sensitivity in lb/in ² at 5 x 10-4 in. plating thickness	Accuracy	Instruments and Description	Measurement Continuous End Point	Calibration of Base Material by	S R E
Dial	.010''	+100 1ъ	80 lb/in ^{2 °} F	+ 10 percent (4) or 1000 lb/in ² whichever is greater	1. Spiral Contractometer (Brenner-Senderoff) Vertical Spiral	x x	Deflection from a given torque	3
Capillary Height	.0375	±80 lb/in ²	530 lb/in ^{2 °} F	+ 5 percent + 5 percent	2. Stresometer (Kushner) 1635: capillary	x x x	Deflection from varying hydro- static pressure	3
End Deflection	.010	±900 lb/in ²	70 lb/in ^{2 °} F	± 1500 lb/in ²	ratio hori- zontal disc	х		
Strain of fibers on back of plated	.008	<u>+</u> 70 lb/in ²	Base material and plating same, no correction	No data	3. Strip test (EOS) Vertical Strip 4. Strip test with strain	x x	Not calibrated Not calibrated	2
strip converted from voltage					gage (EOS) Vertical Strip			

`

	Measurement Continuous End Point	Calibration of Base Material by	Setup Calibration Readout-time Estimate	Instrument Errors and Disadvantages
ter)	x x	Deflection from a given torque	30 min	 1.a. Gear train friction b. Readout accuracy c. Masking d. Vacuum metal finishes not pratical e. Calibration and setup time long
	x x x x	Deflection from varying hydro- static pressure	30 min	Acts as thermometer, accurate temperature control required b. Calibration and setup time long
:	x	Not calibrated	5 min	3.a. Readout accuracy poor b. Thickness uniformity poor c. Not measured at bath temperature d. Point reading
tain	×	Not calibrated	20 mín	 4.a. Masking b. Thickness uniformity poor c. Vacuum metal finishes not practical d. Expensive electronic readout equipment necessary.



TABLE III STRESOMETER TESTS

							SIRESUMETER	10010		
	Test	Date (1)	Tank	Bath	Temperature	Shielding to improve CD distribution	Anode(s)	Disc Thickness	Surface (2)	DC/AC Voltage
. [1	1-30-62	14	SNSR	100.4 ⁺ :1 °F	No	3 boxed oval	.0375"	Cu	2.7-00
	2	1-31-62	14	"	100.4 ^{+.0} ° _F	Yes	11	.005	Br	
,	3	1-31-62	14	11	100.4-0 °F	No	ıı	.003	Br	4.200
ļ	4	2-1-62	14	"	100.4 ^{+.04} °F	Yes	"	.002	Br	6.45+:03
i	5	2-1-62	14	"	100.4 ⁺ :2 °F	Yes	н	.003	Br	6.40+:05
'	6	2-2-62	12	SN	100.4 ^{+.2} °F	Yes	Nickel Disc	.002	Ag-Br	6.3>
	7	2-5-62	12	11	90.9 ^{+.5} °F	Yes	11	.005	Ag-Br	4.1 → 3.5
•	8	2-5-62	12	"	90.9 [±] .0 °F	Yes	11	.005	Ag-Br	3.6-0.5
	9	2-7-62	12	"	92.0 ^{+.5} _F	Yes	"	.005	Br	1.9/5.5
	10	2-7-62	12	11	92.3 ^{+.4} °F	Yes	"	11	Br	3.7-0.0
'	11	2-8-62	12	"	91.8 ⁺ .1 °F	Yes	"	"	Br	1.9/5.5
	12	2-8-62	12	**	92.0 ⁺ :5 °F	Yes	11	11	Ag-Br	3.0 05/4.5
	13	2-8-62	12	**	92.0 <mark>+.0</mark> °F	Yes	"	"	Ag-Br	3.65
	14	2-9-62	12	**	91.8 ⁺ .4 °F	Yes	11	"	Ag-Br	2.9+.8/4.5
	15	2-14-62	14	SNSR	89.6 ^{+1.2} °F	Yes	11	11	Ag-Br	4.8 [±] 0
	16	2-15-62	14	"	90.0 ^{+2.3} °F	Yes	11	"	Br	3.8 2
	17	2-15-62	14	"	90.0 ^{+3.0} °F	Yes	11	11	Br	3.5+.3
	18	2-16-62	12	SN	91.0 ⁺³ _{-0.2} °F	Yes	ti .	"	Br	2.6+:4
	19	2-20-62	12	"	120.0 ⁺¹ :1 °F	Yes	11		Br	2.84
	20	2-22-62	12	"	99.15 ^{+.4} °F	Yes	11	11	Br	2.7-:35
	21	2-22-62	12		109.95 ^{+1.8} ° _F	Yes	**	11	Br	3.8+:3
	22	2-23-62	14	SNSR	90.1-1:3	Yes	Disc	.005	Br	4.012
	23	2-26-62	13	SNSR	90	Yes	Disc	.005	Ag-Br	

⁽¹⁾ Tests prior to 1-30-62 were run with EOS strip tester

⁽²⁾ Cu = copper; Br = brass shim stock; Ag-Br- Vacuum coated silver over brass

⁽³⁾ ASF = Amperes/ft²

	ASF (3)	Purpose of the Test	Stress Measured	Range of Stress in first .0001"	Remarks
	30	Determine procedures for Stresometer	-3,000 lb/in ²	-35,000 lb/in ²	Test invalid - poor shielding technique
	30	Compare sensitivity vs. disc thickness	10,600	15,-26,-18,000	Erratic initial stress
	30	Compare sensitivity vs. disc thickness	14,600	65-40,000	Poor shielding - test invalid
	30	Compare sensitivity vs. disc thickness	29,200	73-64,000	Uncorrected for the effect of diaphragm stresses
	30		27,200	52-40,000	Graphing Street
	30		119,900	171-130,000	Test invalid; anode highly polarized technique
5	10-7 1/2		9,000	16-22,000	Compare Ag with Brass
	7.5	Compare Ag and Brass; DC and AC effects	Thickness too small for accurate reading	40-15,000	
	7.5	Compare Ag and Brass; DC and AC effects	Thickness too small for accurate reading	14-33-24,500	Erratic initial stress
	7.5	Compare Ag and Brass; DC and AC effects	Thickness too small for accurate reading	32 -> 22,000	
	7.5	Compare Ag and Brass; DC and AC effects	Thickness too small for accurate reading	16-28-24,000	Erratic initial stress
	7.5	Compare Ag and Brass; DC and AC effects	Thickness toosmall for accurate reading	20 -> 15,000	
	7.5	Compare Ag and Brass; DC and AC effects	Thickness too small for accurate reading	5 → 15,000	
	7.5	Compare Ag and Brass; DC and AC effects	Thickness too small for accurate reading	10-20-16,000	Erratic initial stress
	15	Determine stress temper- ature profile	16,800 lb/in ²	No data in this range	
	10	Determine stress temper- ature profile	2,500	No data in this	
	10	Duplicate reading No. 16	2,400	No data in this	Close (-4 percent) agreement
	7.5	Determine stress temper- ature profile	14,800	No data in this	with 16 Cleaning of brass discs changed to 25 percent-42° Baume nitric
İ	7.5	Determine stress temper- ature profile	7 ,400	No data in this	73 herceut-47. Raume uitric
	7.5	Determine stress temper- ature profile	11,800	range → 20,000	Close correlation to log stress vs
	7.5	Determine stress temper- ature profile	9,600	No data in this	temperature relationship, see Fig. 2
	10	Duplicate Test 17	15,300	No data	Does not duplicate No. 16 and 17 would expect rise in Stress with
	7.5	Determine stress temper⇔ ature profile	-2,000	No data	time. Other tests indicate a change in bath composition or effect of nitric pickle.



TABLE IV

COMPARISON OF SUPERIMPOSED AC OVER DC CURRENT VERSUS DC CURRENT

SUBSTRATE
BRASS
OVER
SILVER
VACUUM
VERSUS
SUBSTRATE
BRASS
AND

Substrate Subs	- 1				AND BRASS		VERSUS VACITO	M SILVER OVER	SUBSTRATE VERSUS VACIUM SILVER OVER BRASS SUBSTRATE	re	
Brass 1.9 ± .02 2.9 .5 amps Brass 3.7 ± .0 - " Brass 1.9 ± .02 2.9 " Vac Silver over Brass 3.0 ± .05 1.5 " Vac Silver over Brass 2.9 ± 0 1.4 "		Test		DC	Ratio	Current	True St at elec	ress* in 10 ³ trodeposited	lbjin. ² thickness	Instantaneous Stress at	dS d Thickness at 1 x 10-4 in.
Brass 1.9 ± .02 2.9 .5 amps Brass 3.7 ± .0 - " Brass 1.9 ± .02 2.9 " Vac Silver over Brass 3.0 ± .05 1.5 " Vac Silver over Brass 3.60 ± .0 - "		NO.		VOILABE	Voltage		.8 x 10 ⁻⁴ in.	1.0 × 10 ⁻⁴ in	$1.2 \times 10^{-4} \text{in.}$	l x 10-4 in in 103 lb/in.	in 10 ⁷ 1b/in. ³
Brass 3.7 ± 0/2 - " 24.1 21.6 18.5 8.0 Brass 1.9 ± 02 2.9 " 26.6 24.0 21.8 11.7 Vac Silver vac Silver surss 3.0 ± 05 1.5 " 26.6 24.0 21.8 11.7 Vac Silver brass 3.6 ± 0 05 " 17.2 15.1 15.6 1.52 16.0 Vac Silver voer Brass 2.9 ± 0 1.4 " 19.1 16.4 14.9 5.8		6	Brass	1.9 ±.02	2.9	.5 amps	26.3	24.3	21.8	10.7	-10.5
Brass 1.9 ±.02 2.9 " 26.6 24.0 21.8 11.7 Vac Silver Over Brass 3.0 ±.05 1.5 " 17.2 15.7 15.0 8.9 Vac Silver Over Brass 3.60 ±.5 " 15.1 15.1 15.6 1.52 16.0 Vac Silver Over Brass 2.9 ± 0 1.4 " 19.1 16.4 14.9 5.8		10	Brass	3.7 + 0	ı	=	24.1	21.6	18.5	8.0	8.8
Vac Silver over Brass 3.0 + .05 over Brass 1.5 " 17.2 15.7 15.0 8.9 Vac Silver over Brass 3.60 + .0 over Brass 5 " 15.1 15.6 1.52 16.0 Vac Silver over Brass 2.9 + 0 over Brass 1.4 " 19.1 16.4 14.9 5.8		11	Brass	1.9 ±.02	2.9	=	26.6	24.0	21.8	11.7	- 8.6
Vac Silver over Brass 3.60 + .0 5 " 15.1 15.6 1.52 16.0 Vac Silver over Brass 2.9 + 0 1.4 " 19.1 16.4 14.9 5.8		12	Vac Silver over Brass	3.005	1.5	=	17.2	15.7	15.0	8.9	8.9
Vac Silver over Brass 2.9 + 0 1.4 " 19.1 16.4 14.9 5.8		13	Vac Silver over Brass		1	=	15.1	15.6	1.52	16.0	+ 0.4
		14	Vac Silver over Brass	2.9 + 0		=	19.1	16.4	14.9	5.8	-10.6

 \star Plaving thickness and moduli ratio effects corrected 5/7 the following relationship: $s_{
m T}$ = $s_{
m T}$

 $S_T = True stress$ where

■ Indicated stress S

Plating thickness ъ

■ Disc thickness u E_c = Young's modulus of electroplated material

 $E_b = Young's modulus of disc material$

Test	Date	Tank	Temperature	Amps	Volts	Amp.Min.	Thickness in 10 ⁻³ in.	Conductive Coating (1)	Res
2-1	12-12-61	14	100°F	.225	1.2+.2	5,43		ChAg	
2-2	12-14-61	14	100	.225	1.6	6.75	.146	Pass Ni	Compressiv
2-3	12-14-61	14	100	.500	2.0	30.	.65	Pass Ni	Near zero bright
2-4	12-17-61	14	100	.500	2.0	26.5		Ch Ag	Bright
2-5	12-22-61		!	'	{			Ch Ag	
2-6	12-22-61			'				Ch Ag	
2-7	1-5-62	2	90	.170	1	1.15	.025	VAg ²	Very bright
2-8	1-5-62	2	90	.170		3.32	.071	VAg ²	Dull
2-9	1-5-62	2	90	.500		3.375	.103	VAg ²	Very bright
2-10	1-5-62	2	90	.650		4.39	.134	VAg ²	Very bright
2-11	1-8-62	14	100	.650		12.7	.386	VAg ¹	Wrinkled- p Ag
2-12	1-8-62	14	100	.650		12.7	.386	VAg ¹	Wrinkled- p
2-13	1-11-62	14	100	.650		12.0	.366	VAg ²	Bright
2-14	1-11-62	14	100	.650		22.1	.674	VAg ²	Bright
2-15	1-19-62	2	100	.650	-	17.2	.525	VAg ²	Wrinkled, co
2-16	1-19-62	2	94	1.00	3.8	12.5	.381	VAg ²	Center brigl
2-17	1-22-62	2	90	1.0	3.8	9.2	.281	VAg ¹	Wrinkled- st rotor?- thir
2-18	1-22-62	2	90	1.14	3.8	4.69	.143	VAg ¹	Wrinkled- st silver

TABLE V (continued)

Results	Profil (2)	Thickne	Backing Stess, type,	ructure surface quality	Adhesive System (4)	Cure °F/Hr	Parting Results
Compressive - Dull		1"	Pyrex	125 emery	826/DEAPA/8	170/2	Difficult
Near zerc stress - and bright							
Bright		1"	Pyrex	125 emery	826/DEAPA/8	200/2	Difficult
		1"	Pyrex	125 emery	828/DTA/8	85/17	Difficult
		.5"	Alum	10 RMS	828/DTA/8	85/18	Moderate
Very bright	± 3/4 λ						
Dull	± 1-1/4λ	1"	Pyrex	125 emery	826/DTA/10	85/88	Failed due to
Very bright	±1λ	1"	Pyrex	125 emery	826/DTA/10	85/88	heat shock Failed due to
Very bright	+1λ	i		1			heat shock
Wrinkled- pin holes in Ag							
Wrinkled- pin holes in Ag							
Bright	± 4 λ	1"	Pyrex	125 emery	828/DEAPA/8	90/16	Difficult
Bright	± 11 λ	1"	Pyrex	125 emery	828/DEAPA/8	90/16	Difficult
Wrinkled, compressive							
Center bright			,				
Wrinkled- short in rotor?- thin silver							
Wrinkled- short- thin							

TABLE V (continued)

nesive vstem 4)	Cure OF/Hr	Parting Results	Master Accuracy	Replica Accuracy/ Irregularities (5)	Difference (6)	Comments
EAPA/8	170/2	Difficult	1/2 cc	1 cv/.5	.5 λ	Chemical silver has strong adhesion
EAPA/8	200/2	Difficult	1/4	3 cv/1	2-3/4 λ	
r a/ 8	85/17	Difficult	1/2 cc	1 cv/40.5	1/2 λ	
FA/8	85/18	Moderate	1/4	1/40.5	3/4 A	Alum thermal expansion may aid parting
rA/10	85/88	Failed due to heat shock				
TA/10	85/88	Failed due to heat shock				
EAPA/8	90/16	Difficult	1/8 cv	1/2/1/2	3/8 _λ	Master warped after
APA/8	90/16	Difficult	2 cv	2 cc	0 λ	parting



TABLE V (continued)

Test	Date	Tank	Temperature	Amps	Volts	Amp.Min.	Thickness	Conductive Coating	Results
2-19	1-24-62	14	90	.9	3.2	12.5	.382	VAg ²	Center Bright
2-20	1-26-62	14	90	.9	3.2	10.0	.329	VAg ²	
2-21	1-31-62	14	90	.3		4.5	.137	Alum Foil	No adhesion
2-22	2-5-62	2	86		2.4	12.9	.395	VAg ²	Short, partly wrinkled
2-23	2-6-62	14	90	.780	5.9	20.6	.678	VAg ²	Bright center, dull outside, wrinkled
2-24	2-6-62	14	93	.7		20.3	.667	VAg ²	Rough, wrinkled on standing
2-25	2-6-62	14	90	.780	3.5	22.6	.69	VAg ²	Matte finish, rough, couldn't read
2-26	2-7-62	14	90	.780	6.3	25.4	.831	VAg ²	Dull, surface washed of when rinsed
2-27	2-8-62	14	90	.780	6.0	690	21.0	VAg ²	Rotor stopped at night badly pitted
2-28	2-9-62	14	90	.780		26.9	.82	VAg ²	Edges lifted on standin no reading
2-29	2-13-62	14	90	.780	6.2	26.1	.857	VAg ²	Dull to bright
2-30	2-13-62	14	90	.780	5.9	25.0	.761	VAg ²	Lifted during plating - to much agitation
2-31	2-21-62	14	90	1.1	7.4	36.8	1.12	VAg ²	Edges slightly raised Slight leakage under sk Poor master cleaning
2-32	2-22-62	14	90	.66	50	38	1.56	VAg ²	Semi-bright back - repl washed off in rinsing

Note: 1. ChAg = Spray chemical silver, Pass Ni = Passivated nickel, VAg = 1 layer vacuum coated silver, VAg = 2 layers vacuum coated silver, Alum foil = Aluminum foil

- 2. Profile variations given in wavelengths of light, λ where λ = 21.6 x 10^{-6} inches
- 3. Backing structures Pyrex = pyrex glass, Alum = aluminum
- 4. Adhesive system = Shell resin/hardener/hardener parts per hundred
- 5. Irregularities in wavelengths of light λ ; cc = concave; cv = convex
- 6. Deviation from perfect replication in wavelengths of light, λ 2110-Q-1



TABLE V (continued)

lts	Profile		Backing Structure ss, type, surface quality	Adhesive System	Cure F/Hr	Parting Results	Master Accuracy
ht	+ 5-1/2λ	1"	Alum Mill finish	828/DEAPA/8	95/111	Skin separated due to holding fixture	
	± 5 λ	1"	Alum Mill finish Light.	828/DEAPA/8	100/55	Skin separated	
ly wrinkled			_	828/DEAPA/8	113/140	Poor adhesion due to shock, SiO over nickel	Distorted
er, dull inkled	± 8-1/4 λ						
kled on							
h, rough,							
ce washed off							
ed at night - d							
d on standing-							
ght	+ 1-1/2 λ	1"	Alum	828/DEAPA/8	113/140	Edges partly lifted du	e to poor
ng plating - tation						adhesion and heat shock	1
tly raised age under skin cleaning	± 1-3/4 λ						
back - replica in rinsing							



.

TABLE VI SUMMARY OF 5-1/2" REPLICAS FROM 6" DIAMETER 14.188" RADIUS SPHERICAL MASTERS

Test	Date	Master No.	Tank	Temp.	Volts	Conductive Coating (1)	Current Density(2)	Current (amps)	Time (hours)	Amp-Hrs.
3-1	12-1-61	251	14 15	100 100	2.1 8.1 ± ·5	SAg	7.5 75	1.3 13	1.2 16.2	1.56 211.1 212.66
3-2	2-2-62	251	14	90	3.7	VAg ²	2.9	.500	1.467	.73
3-3	2-5	251	14	90	4.5	VAg ²	21.3	3.75	20.5	76.9
3-4	2-6	253	14	90	3.9	VAg ²	20.3	3.5	18.33	64.2
3-5	2-7	252	14	90	5.0	VAg ²	20.3	3.5	18	63
3-6	2-8	252	14	90	5.0	VAg ²	21.3	3.75	.83	6.25
3-7	2-13	251	14	90	3.6	vag ²	21.3	3.75	17.63	66.1
3-8	2-23	251	14	90	4.6	vag ²	2.2	3.8	65.0	24.7
3-9	2-27	253	13	90	2.4	VAg ²	10.1	1.75	1.25	2.19
3-10	2-27	252	13	90	3.0	VAg ²	14.5	2.5	16	40
	ĺ	,								
					<u></u>					

Note: 1. SAg = Sprayed silver, VAg² = vacuum silver (two layers)

- 2. Current density in amperes/ft²
- 3. Thickness in .001 inches = $\frac{\text{ampere hrs x 90 percent efficiency}}{21.04 \text{ gm/mil sq ft}^2}$
- 4. See Fig. 3 for nomenclature
- 5. CD = Current density

US SPHERICAL MASTERS

	Current	Time		m ()				Anode	Relationships	(4)			Res
(2)		(hours)	Amp-Hrs.	Thickness (3) (10 ⁻³ inches)	θ	а	ъ	е	Anode 1	Gas Vents	f	Anode 2	
	1.3 13	1.2 16.2	1.56 211.1 212.66	59.5	0°					No	16"	3 3-1/2" ovals 9" long	Parting extremely diffic Crack appeared after par used to provide conducti
	.500	1.467	.73	.2	45°			1"	3/16" x 6" centered	11	16"		Center 2"; bright, outer
	3.75	20.5	76.9	21.5				İ			1		
	3.5	18.33	64.2	18.0	45°			1"	3/16" x 6"	11	16"	2 3-1/2" ovals 9" long	Outer edges lifted and d shorted meter
	3.5	18	63	17.6	45 ⁰			1"	3/16"x2-1/2"	11	16"	2 3-1/2" ovals 9" long	Edges lifted
	3.75	.83	6.25	17.5	45°	1/2"	4-1/2"	1''	3/16"x2-1/2"	Yes	16"	2 3-1/2" ovals 9" long	Edges under shield were replica slated to study
	3.75	17.63	66.1	18.5	45°	1/2"	5"	1"	3/16"x2-1/2"	17	16"	2 3-1/2" ovals 9" long	Replica slated for thin
	3.8	65.0	24.7	69.1	45 ⁰	1/2"	5"	1"	3/16x2-1/2"	"	16"	2 3-1/2" ovals 9" long	Edges slightly distorted
	1.75	1.25	2.19	.78	45°	1/2"	5"	1"	3/16"x2-1/2"	"	16"	2 3-1/2" ovals 9" long	Orange peel, fair edge d
	2.5	16	40	14.3	45°					"	16"	2 3-1/2" ovals 9" long	CD too low; compressive
					45°					ш	16"	2 3-1/2" ovals 9" long	Very good edge
		l											



TABLE VI (continued)

		Results and Remarks	Conclusions (5)
f	Anode 2		
16"	3 3-1/2" ovals 9" long	Parting extremely difficult; used heat shock, air, ultrasonics. Crack appeared after parting; replica highly disturbed; 2 tanks used to provide conductive skin for high CD in second tank.	High CD plating impractical; optical distortion caused by surface defects is great at high CD and heavy deposits. Sprayed silver is too adhesive.
16"		Center 2"; bright, outer edges distorted	Overcompensated center current density deficiencies
16"	2 3-1/2" ovals 9" long	Outer edges lifted and disclosed ac current effects from shorted meter	Insulate motor
16"	2 3-1/2" ovals 9" long	Edges lifted	
16"	2 3-1/2" ovals 9" long	Edges under shield were highly compressive and distorted; replica slated to study shielding and for Rh plating.	Use smaller shield, further away.
16"	2 3-1/2" ovals 9" long	Replica slated for thin film replication studies	
16"	2 3-1/2" ovals 9" long	Edges slightly distorted	
16"	2 3-1/2" ovals 9" long	Orange peel, fair edge distortion	Heavy deposit causes surface dimpling
16"	2 3-1/2" ovals 9" long	CD too low; compressive center	
16"	2 3-1/2" ovals 9" long	Very good edge	

TABLE VI (continued)

		Results and Remarks	Conclusions (5)
f	Anode 2		
16"	3 3-1/2" ovals 9" long	Parting extremely difficult; used heat shock, air, ultrasonics. Crack appeared after parting; replica highly disturbed; 2 tanks used to provide conductive skin for high CD in second tank.	High CD plating impractical; optical distortion caused by surface defects is great at high CD and heavy deposits. Sprayed silver is too adhesive.
16"		Center 2"; bright, outer edges distorted	Overcompensated center current density deficiencies
16"	2 3-1/2" ovals 9" long	Outer edges lifted and disclosed ac current effects from shorted meter	Insulate motor
16"	2 3-1/2" ovals 9" long	Edges lifted	
16"	2 3-1/2" ovals 9" long	Edges under shield were highly compressive and distorted; replica slated to study shielding and for Rh plating.	Use smaller shield, further away.
16"	2 3-1/2" ovals 9" long	Replica slated for thin film replication studies	
16"	2 3-1/2" ovals 9" long	Edges slightly distorted	
16"	2 3-1/2" ovals 9" long	Orange peel, fair edge distortion	Heavy deposit causes surface dimpling
16"	2 3-1/2" ovals 9" long	CD too low; compressive center	
16"	2 3-1/2" ovals 9" long	Very good edge	

SUMMARY OF

Test	Date	Replica Test No.	Temp	Volts	Base Metal	Current Density Amps/ft ²	Repl Sizo Dia.		Amps
4-0	2-16-62	2-26	105 ⁰ F	2	Ag	10.6	2-5/8"	.0376"	.400
4-1	2-16	4-1	105	2	Rh+Ag	10.6	2-5/8	.0376	.400
4-2	2- 16	2-7	105	2	Ni	10.6	2-5/8	.0376	.400
4-3	2-16	3-5	105	2	Ni	9.6	5-5/8	.1727	1.66
4-4	2-19	(1)	105	2	Ag	.3	2-1/2	.0341	.100
4-5	2-20	(1)	105	2	Ag	44	2-1/2	.0341	.5-1.5
4-6	2-20	(1)	105	2	Ag	44	2-1/2	.0341	1.5
4-7	2-20	(1)	105	2	Ag	5.9	2-1/2	.0341	.2
4-8	2-21	4-1	105		Rh				.350
4-9	2-26	(1)	105		Ni	10.3	2-1/2	.0341	1.2
4-10	2-26	м-н-11-61	105		Ni	35	5-5/8	.1727	600
4-11	2-26	3-4	105		Ni	35	5-5/8	.1727	6.00
4-12	2-26	3-7	105	ļ	Ni	35	5-5/8	.1727	6.00
4-13	2-26	3-8	105		Ni	35	5-5/8	.1727	6.00

Notes: 1. New samples etched from nickel sheet

2. Thickness in 10^{-6} in. = $\frac{\text{amp. min.} \times 70 \text{ percent efficiency}}{1.375 \text{ amp min/area in ft}^2}$



TABLE VII
MMARY OF RHODIUM PLATED REPLICAS

Amps	Min.	Amp-	Thickness in 10 ⁻⁶ in (2)	Agitation	Results
.400	1.1	.44	6.0	Hand	Pitted, black spots due to hydrogen, no rinse after acid dip
.400	1.1	.44	6.0	Hand	Used reverse current etch on ${\rm H_2SO_4}$ solution; still bad
.400	1.1	.44	6.0	Hand	Good, bright
1.66	1.1	1.826	6.0	Hand	Good, bright
.100	1.5	.150	2.2	Stirrer	Spotty, pealing at edges
.5-1.5	1.1	1.1	16	Stirrer	Hazy, rhodium sealing
1.5	1.1	1.65	24	Stirrer	Cloudy and spotty
.2	5	1.0	15	Stirrer	Cloudy and spotty
.350	1.1	.385	5.7		Cloudy and spotty
1.2	.2	.24	3.5	None	Good, bright
600	2	1.2	3.5	None	Good, bright
6.00	.2	1.2	3.5	None	Good, bright
6.00	.2	1.2	3.5	None	Good, bright
6.00	.2	1.2	3.5	None	Good, Bright